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Structural and vibrational properties of CVD grown few layers MoS_2 on catalyst free PAMBE grown GaN nanowires on Si (111) substrates

Mansi Agrawal [a,*], Anubha Jain [b], Vishakha Kaushik [a], Akhilesh Pandey [b], B.R. Mehta [a], R. Muralidharan [c]

[a] Department of Physics, Indian Institute of Technology Delhi, Hauz Khas, New Delhi, 110016, India
[b] Solid State Physics Laboratory, Timarpur, Delhi, 110054, India
[c] Centre for Nano Science and Engineering, Indian Institute of Science, Bangalore, 560012, India

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ABSTRACT

In the present work, catalyst free GaN nanowires were grown on Si (111) substrates by plasma assisted molecular beam epitaxy followed by chemical vapour deposited few layers MoS_2. The morphology of the GaN nanowires and MoS_2/GaN were studied using scanning electron microscopy. The vibrational properties of MoS_2/GaN nanowires/Si (111) substrates were analysed using Raman spectroscopy. The difference of 24.8 cm^{-1} between E_{2g} and A_{1g} Raman peaks confirmed few layers of MoS_2. High Resolution X-ray diffraction results in accordance with Raman results also indicated the growth of few layers of MoS_2 on GaN nanowires grown on Si (111) substrates. As a result MoS_2/GaN nanowires heterojunction with their exceptional structural and vibrational properties as studied in the present work can have great potential for future optoelectronic devices.

1. Introduction

The integration of technologically advanced III-Nitrides and 2D materials is of great interest for wide range of applications such as photodetectors, field effect transistors and photo catalytic applications [1–3]. Heterostructures based on GaN find applications in several fields ranging from high power devices, optoelectronic devices, military applications and high frequency devices [4–11]. 2D metal dichalcogenides especially MoS_2 have opened a new route in the recent years and generated interest as they are potential candidates for optoelectronic devices like photodetectors, lithium ion batteries, spintronics, field effect transistors, supercapacitors and energy storage devices [12–15]. The moderate mobility of the charge carriers, direct energy bandgap, strongly bound excitons and high surface area of 2D materials allow applications in electronic devices [16]. In addition 2D materials are thermally stable and have a negligible lattice mismatch with III-nitrides [17]. Recently Moun et al. have reported on MoS_2/GaN heterojunction diode based photodetector with a high sensitivity of 10^5 A/W and detectivity of 10^{14} Jones 18]. Chen et al. reported $MoSe_2$/GaN heterojunction diode can be utilised for optoelectronic applications with a photovoltaic emission at 850 nm for energy harvesting [19]. Goel et al. reported on the integration of MoS_2 with GaN for ultraviolet photodetector applications with a spectral responsivity of 10^3 A/W20. Zhang et al. reported that integration of MoS_2 with GaN can be utilised as a photocatalyst for water splitting applications using solar energy after nitridation [21]. Desai et al. have assessed the rectifying diode characteristics of vertical device based on 2D MoS_2 on n-type GaN and obtained photovoltaic responsivity suitable for optoelectronic applications [22]. Therefore, the integration of III-Nitrides and 2D materials such as MoS_2 needs to be studied in detail. Although there are few reports of MoS_2 on GaN thin films but there not much reports on few layers of two dimensional MoS_2 on GaN nanowires. Thus, it becomes essential to study the properties of MoS_2/GaN nanowires.

Group III-Nitrides have attracted significant attention in the recent years due to their direct band gap (3.4 eV) in comparison to silicon, high electron mobility, high saturation velocities (~2×10^7 cm/s), large breakdown fields (~3 MV/cm), chemical stability and ability to operate at high temperature [23,24]. Recently, GaN light emitting diodes (LEDs) combining with quantum dots have also attracted significant attention such as Guan

* Corresponding author.
E-mail address: mansiagra2000@gmail.com (M. Agrawal).

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et al. have fabricated white LEDs with an efficiency of 40.6 lm W^{-1} by combining silica-coated $CsPbBr_3$ quantum dots with In–Ag–Zn–S quantum dots on InGaN blue LED, Zang et al. have reported an improvement of 58.4% in the output power by using roughened ZnO and microhole arrays on InGaN LEDs, Han et al. have reported on $CsPb_2Br_5$ nanoplatelets on blue GaN LED for green LEDs with an efficiency of 34.49 lm W^{-1} and photodetectors applications and Yan et al. have reported enhanced efficiency of green LEDs by combining $CsPbBr_3$ quantum dots with InGaN blue LED chip [25–28]. Due to the lack of native substrate, the most commonly used substrates for the growth of these materials are SiC, sapphire and silicon [29]. Silicon is the most commonly used substrate for the growth of GaN films because of low cost possibility of realizing vertical devices with Si as substrates in comparison to free-standing substrates [30,31]. However, GaN layers suffer from threading dislocations (of the order of ~10^9 cm^{-2}) which result in poor performance of the devices and surface related defects owing to large thermal and lattice mismatch between the substrates and GaN [32–34]. III-nitride semiconductor epitaxial films can be grown by various techniques like chemical vapour deposition, metal organic chemical vapour deposition, pulsed laser deposition and plasma assisted molecular beam epitaxy [35–38]. However, plasma assisted molecular beam epitaxy is the preferred choice because of controlled growth rates in ultrahigh vacuum thereby yielding high quality, epitaxial III-Nitride films [39].

Molybdenum disulphide is a metal dichalcogenide with a two dimensional layered structure of the form S–Mo–S in which the atomic planes of molybdenum is sandwiched between the atomic planes of sulphur atoms on either side. Although there exists a strong covalent bond between the Mo and S atoms but there is a weak van der Waal interaction between the layers [40]. It is a direct bandgap material with a bandgap of 1.9 eV unlike its bulk counterpart which is an indirect bandgap semiconducting material with a bandgap of 1.2 eV due to which 2D MoS_2 is a promising material for many optoelectronic devices [41]. Another added advantage of this two dimensional material is better quantum confinement due to the increase in surface to volume ratio as compared to bulk MoS_2.

In the present study GaN nanowires were grown using plasma assisted molecular beam epitaxy on Si (111) substrates. The main advantage of growing the nanowires by this technique is that the growth can be initiated without any catalyst [42]. GaN nanowires also offer better quantum confinement because of higher surface to volume ratio unlike the GaN thin films. It has been reported that catalyst free GaN nanowires grown by plasma assisted molecular beam epitaxy are of high structural quality as they are free from any residual strain and have reduced structural defects in comparison to GaN nanowires grown in the presence of a catalyst [43,44]. Chemical vapour deposition is used to grow single crystalline, large area, layered transition metal dichalcogenides such as MoS_2 [45]. In comparison to other techniques for layered MoS_2 growth like mechanical and chemical exfoliation, lithium intercalation, wet chemical methods, CVD prepared films are stoichiometric and have low density of defects [45–49]. In the present study, few layers of molybdenum disulphide were deposited on GaN nanowires by chemical vapour deposition as this method gives high quality of MoS_2. The structural and crystallographic properties of the GaN nanowires and MoS_2/GaN nanowires were investigated using scanning electron microscopy and high resolution electron diffraction respectively. Raman spectroscopy is utilised to study the vibrational properties and number of layers of MoS_2 in the sample.

2. Experimental details

The growth of GaN nanowires was performed in SemiTEq, STE3N2 molecular beam epitaxy system using VEECO radio frequency plasma source. 3 inch, n-type and low resistivity epi-ready Si (111) substrates were used for the growth experiments. The substrate was initially thermally degassed in-situ at 1170 °C to remove any native oxide. The substrate temperature was recorded using MODLINE IRCON and MIKRON pyrometers. 7 × 7 reconstruction was observed at the substrate temperature of 830 °C as shown in Fig. 1(a). The substrate temperature was again increased to 1170 °C followed by the nitridation of the substrate using 500 W plasma power and 1.5 sccm of nitrogen gas flow for 15 min. A clear streaky 1 × 1 in-situ reflection high energy electron diffraction pattern was observed after nitridation as shown in Fig. 1(b). Following nitridation the growth of nanowires was carried out by ramping down the substrate temperature to 780 °C. For GaN growth the shutter of Ga metal Knudsen cell was opened at the beam equivalent pressure of 6.37×10^{-5} Torr and nitrogen flow rate was kept at 1.5 sccm at a plasma power of 450 W. Initially there was no change in the streaky RHEED pattern but after few minutes of GaN growth the streaks converted to spotty like patterns as shown in Fig. 1(c). For catalyst free growth of nanowires crystalline silicon nitride layer formed due to the nitridation of the substrate which acts as nucleation sites for the growth of the nanowires. The thickness of GaN was approximately 450 nm as measured in-situ by laser interferometer using a 633 nm wavelength red laser.

A low-pressure chemical vapour deposition (LPCVD) technique was used for the synthesis of MoS_2 utilizing a two-temperature zone furnace with a quartz tube, an upstream zone for the evaporation of sulphur (S_2) powder, and a downstream zone for solidification of reactive species. Moly-oxide and sulphur were used as the growth precursors and the deposition temperature was optimized to be kept around 800 °C for the reaction between gaseous precursors to take place.

The morphology of GaN nanowires and MoS_2/GaN nanowires was analysed by field emission scanning electron microscopy (FESEM). X-ray pole figure measurements of GaN nanowires for (0002) and (101) reflections were performed using Panalytical X'Pert Pro MRD HRXRD system in which parabolic mirror was placed in the incidence geometry and 0D mode of Pixel detector was used on the detector side. Grazing Incidence X-Ray diffractogram (GIXRD) scan was performed to identify GaN and MoS_2 phase. For the measurements the angle of incidence ~2° was fixed from the sample surface and the detector was scanned from (30–80°). Raman spectroscopic measurements were performed at room temperature by Horiba Jobin Yvon LABRAM HR Evolution Confocal Micro-Raman spectrometer system using visible (532 nm) laser excitation.

3. Results and discussions

The length and diameter of GaN nanowires depends upon the growth parameters namely the growth temperature, III-V ratio and the deposition time. Mata et al. have reported the length of the nanowires is nearly constant and the diameter decreases linearly with growth temperature in the range 770 °C–790 °C [50]. It has been reported that when the deposition time is greater than the nucleation time then the relation between length and diameter of the nanowires can be explained by diffusion induced mechanism [51]. Fig. 2 shows the scanning electron microscopy results of GaN nanowires on Si (111) substrates. The plan view and cross-sectional view of the nanowires are shown in Fig. 2(a) and (b) respectively. The results clearly show the formation of GaN nanowires on Si (111) substrates. The diameter of the nanowires ranges from 20 nm to 90nm and the length of the nanowires is ~450 nm. The variation in the length and diameter of the nanowires can be ascertained to the fact the nanowires were grown without using a catalyst at a temperature of 780 °C and the deposition time was 1 h. The results also

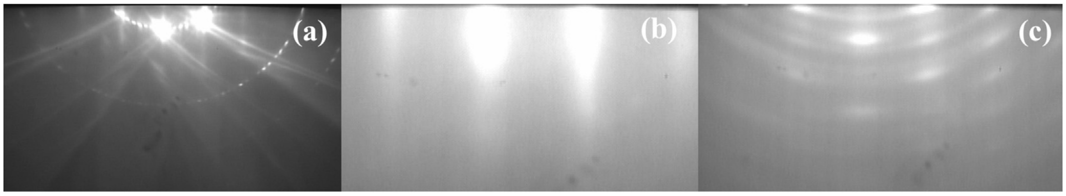

Fig. 1. RHEED patterns (a) 7 × 7 reconstructuction after thermal degasing, (b) after nitridation of Si (111) substrates, (c) during the growth of GaN nanowires on Si (111) substrates.

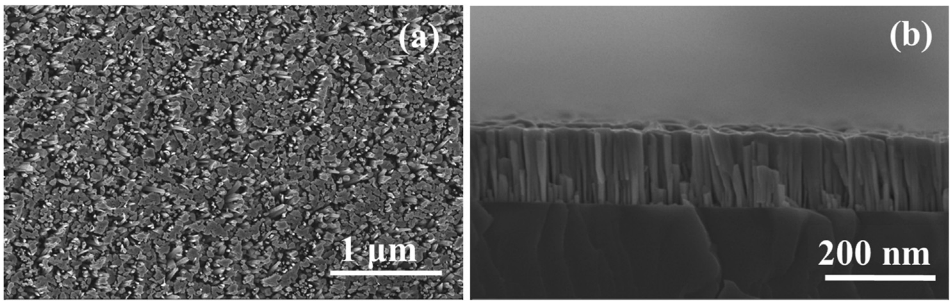

Fig. 2. FESEM images of GaN nanowires on Si (111) substrates (a) Plan view, (b) Cross-sectional view.

indicate that the nanowires are highly dense and of good structural quality growing perpendicular to the substrates, in consistency with the literature [52].

High resolution X-ray diffraction measurements were carried out to study the crystallographic structure. X-ray pole figure measurements were performed for GaN nanowires on Si (111) substrates to determine the tilt in the nanowires along (0002) GaN reflection. The tilt angle (χ) was varied from $-48°$ to $+48°$ at fixed 2Θ angle and azimuth (Φ) scans were measured. The pole figure of GaN (0002) reflection is shown in Fig. 3(a) which shows only one maximum spot at ~$\Phi = 90°$ which indicates that the nanowires are aligned perpendicular to Si (111) substrates and are not randomly oriented in any other direction. These results are in agreement with SEM results discussed above. Fig. 3(b) shows the pole figure measurements at (101) reflection. The six equally spaced maximum intensity spots (~60°) reflect that the GaN nanowires show six-fold symmetry and crystallise in the wurtzite phase. These spots are slightly tilted ~2-3° towards $\Phi = 90°$ side. This indicates that the grown nanowires are almost perpendicular to Si (111) substrate with a slight tilt of 2-3°.

Fig. 4 displays the high resolution x-ray diffraction patterns of the GaN nanowires on Si (111) substrates after MoS_2 deposition. I-

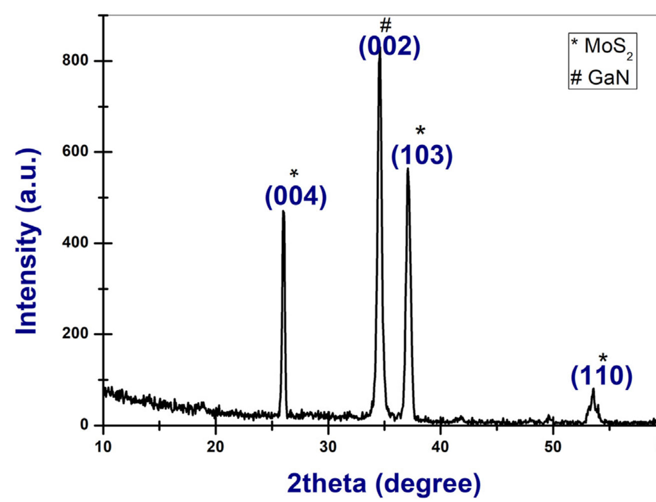

Fig. 4. HRXRD results of few layers of MoS_2/GaN nanowires on Si (111) substrates.

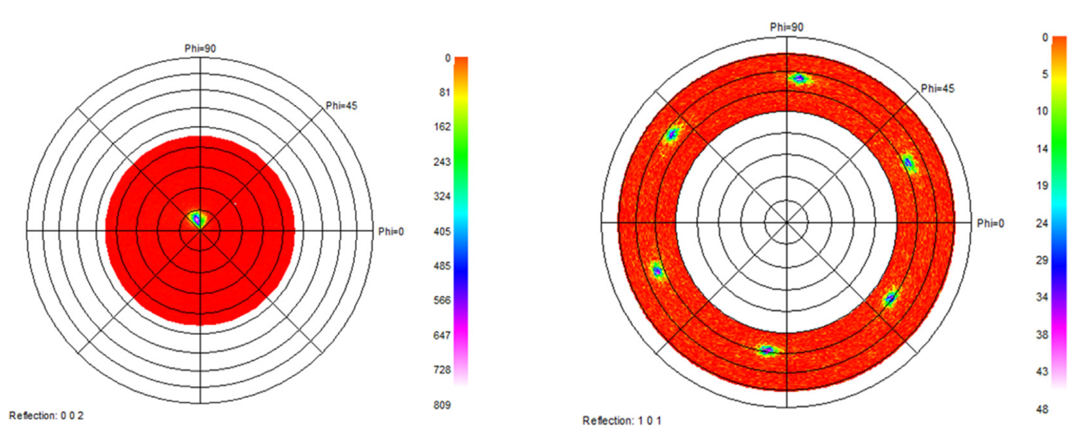

Fig. 3. X-ray pole figure results of GaN nanowires on Si (111) substrates (a) along GaN (002) reflection, (b) along (101) reflection.

2Θ scans at grazing angle were performed to observe the results. The peaks at 25.96°, 37.11° and 53.52° can be attributed to (004), (103) and (110) planes of MoS_2. The absence of the peak related to the (002) plane of MoS_2 may be attributed to the few layers of MoS_2 on GaN nanowires. The diffraction peaks were indexed using JCPDS card no. 37–1492 [20]. The peak at 34.67° corresponds to (0002) plane of GaN. It also reflects that the nanowires are crystalline in nature and are oriented along <0001> direction. A sharp peak of GaN is observed due to an approximate thickness of 450 nm of the nanowires in comparison to only few layers of MoS_2. In addition, it was observed that (004) peak corresponding to MoS_2 has a narrow full width half maximum indicating that MoS_2 grows along c-axis and is highly crystalline.

Scanning electron microscopic results of MoS_2 on GaN nanowires/Si (111) substrates are shown in Fig. 5. The measurements were performed at an accelerating voltage of 10 kV. SEM results clearly indicate the formation of GaN nanowires decorated with triangular shaped MoS_2 domains on top as indicated in the figure. It has been reported in literature that MoS_2 layers grown by chemical vapour deposition exhibit a triangular domain structure also evident from the results shown above [45].

Raman spectroscopy was used to study the structural and vibrational properties of MoS_2 on GaN nanowires on Si (111) substrates. The optoelectronic properties of MoS_2/GaN heterostructures are strongly influenced by the number of layers of MoS_2 domains. Raman spectroscopy is one of the non-destructive efficient tools to determine the number of layers in MoS_2. The Raman spectra of MoS_2 mainly consists of two Raman active modes namely the E_{2g} (383 cm^{-1}) mode and A_{1g} mode (409 cm^{-1}) [53]. The A_{1g} mode also called the out-of-plane mode is associated with the out-of-plane vibration of sulphur atoms. The E_{2g} mode or the in-plane mode arises because of the vibration of molybdenum and sulphur atoms in opposite directions. The number of layers is a function of frequency difference between A_{1g} and E_{2g} modes. A_{1g} mode arises because of the vibration of sulphur atoms and its frequency depends on the interaction of adsorbates with MoS_2 [54]. It has been reported that the frequency of A_{1g} mode shows a blue shift due to enhanced electron phonon coupling while that of E_{2g} shows a red shift because of decrease in long range Columbic interaction due to increase in the dielectric screening with increase in the number of layers [55,56]. With increasing number of layers of MoS_2, A_{1g} mode blue-shifts owing to larger accumulated restoring force. However, the red-shifting of E_{2g} mode can be attributed to the surface effect due to the forces present at the surface of the thin film. Therefore, as the number of layers of MoS_2 increases, the frequency of E_{2g} mode decreases while that of A_{1g} mode increases, thereby, the frequency difference between these two Raman modes increases.

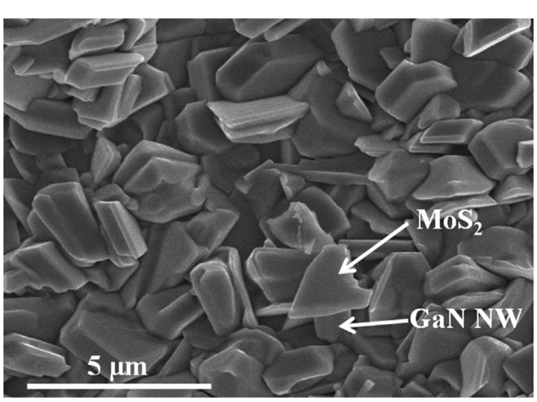

Fig. 5. SEM results of few layers of MoS_2/GaN nanowires on Si (111) substrates.

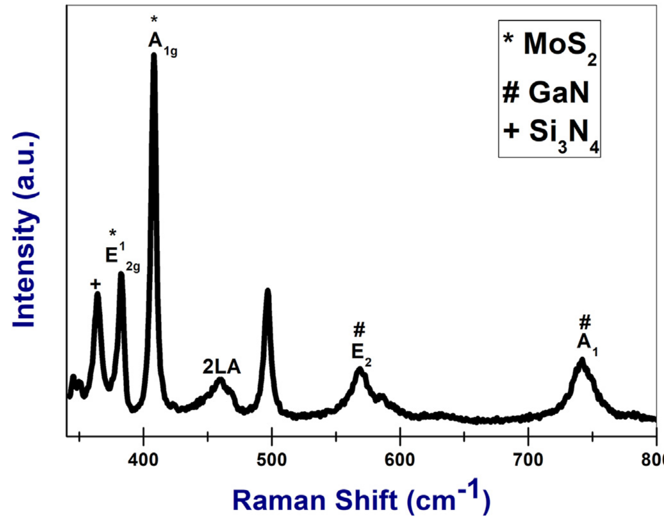

Fig. 6. Raman spectra of MoS_2/GaN nanowires on Si (111) substrates.

Fig. 6 shows the Raman spectra of MoS_2/GaN nanowires/Si (111) which validates the vibrational properties of MoS_2-GaN nanowires heterostructures. The E_{2g} (in-plane) and A_{1g} (out-plane) peaks for MoS_2 were observed at 383.2 cm^{-1} and 408 cm^{-1} respectively. The difference of 24.8 cm^{-1} between the peaks confirms few layers of MoS_2 on GaN nanowires [18,55]. The large frequency difference can be ascertained to the fact that in this study 5–6 layers of MoS_2 are grown on GaN nanowires. There are few bands which are the combination of coupling with the longitudinal acoustic mode LA (M). The asymmetric peak observed at 460 cm^{-1} can be assigned to second order 2LA (M) mode. The frequency of longitudinal acoustic mode is 233 cm^{-1} [53,54]. In addition Frey et al. [56] have reported that the 2 LA (M) mode further splits into multiple peaks. Li et al. [40] have reported that one of the peaks at 440 cm^{-1} due to oxy-sulfide species can be observed only in ultrathin MoS_2 samples but since in our case the thickness of MoS_2 is limited to few layers we did not observe this peak in our measurements. Thus, the peak at 460 cm^{-1} in our results is attributed to 2LA (M) mode. The peaks observed at 568 cm^{-1} and 743 cm^{-1} corresponds to E_2 high and A_1 (LO) vibrational modes in GaN. The frequency difference $\omega - \omega_0$ in the E_2 high peak positions of GaN nanowires and stress free GaN can be used to calculate the biaxial stress (σ) in the nanowires using the relation $\omega - \omega_0 = -k_s\sigma$ where, $k_s(= 2.56\ cm^{-1}/GPa)$ is the proportionality constant [57]. The negative sign in the above equation indicates compressive stress. The value of σ for GaN nanowires is found to be −0.16 GPa indicating the relaxation of compressive stress in the nanowires. The Raman peak observed at 364 cm^{-1} can be attributed to Si_3N_4 [58] which is formed after nitridation of Si (111) substrates during the growth of GaN nanowires. Thus, our results suggest that MoS_2-GaN nanowires are potential candidates for future optoelectronic device applications.

4. Conclusions

The structural and vibrational properties of chemical vapour deposited few monolayers of MoS_2 on GaN nanowires grown by plasma assisted molecular beam epitaxy on Si (111) substrates are investigated. FESEM results indicate that GaN nanowires grow perpendicular to the Si (111) substrates. The SEM results also confirm that GaN nanowires are decorated by triangular shaped MoS_2 domains. X-ray pole figure measurements show that the nanowires have a wurtzite structure and oriented perpendicular to

the substrate. The (004) diffraction peak at 25.96° reflects the formation of two dimensional MoS_2 layers on GaN nanowires. The difference between A_{1g} and E_{2g} Raman peaks is 24.8 cm^{-1} which further confirms few monolayers of MoS_2. Furthermore, the 2LA (M) is observed at 460 cm^{-1}. Thus, our results show that the integration of GaN nanowires with two dimensional MoS_2 paves the way for futuristic optoelectronic and high power devices.

CRediT authorship contribution statement

Mansi Agrawal: Conceptualization, Methodology, Validation, Formal analysis, Writing - original draft. **Anubha Jain:** Investigation, Resources, Writing - review & editing. **Vishakha Kaushik:** Resources, Writing - review & editing. **Akhilesh Pandey:** Resources, Visualization, Writing - review & editing. **B.R. Mehta:** Supervision, Conceptualization, Writing - review & editing. **R. Muralidharan:** Supervision, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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